X-RAY DIFFRACTION STUDY OF PHASE FORMATION AND GROWTH IN NITROGEN IMPLANTED IRON: TEMPERATURE EFFECTS

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INTRODUCTION

One of the dominant mechanisms by which ion implantation hardens structural materials is the formation of second phase precipitates.[1,2] Extensive transmission electron microscope (TEM) work has demonstrated that nitrogen implanted iron contains microscopic grains of a variety of stable and meta-stable iron nitride phases.[3,4,5,6] Typically, these grains are quite small and are identified from densitometer traces of electron diffraction patterns which reveal a rich array of minute grains with only slightly different lattice parameters. Thus, it is often difficult to resolve adjacent diffraction peaks and obtain quantitative information.

X-ray diffraction (XRD), traditionally a bulk analysis technique, has rarely been applied to ion implanted structural materials because of the typically shallow surface layers (≈ .1µm) involved. However, by using x-rays which are strongly absorbed by the host material along with a crystal monochromator to curtail the resulting fluorescence, it is possible to obtain surface information from XRD.[7] The most prominent feature of our measured XRD patterns is the appearance of sharp iron nitride lines corresponding to large crystallites of Fe_2N and Fe_4N. X-ray diffraction, which is not sensitive to small grains but offers extremely well-resolved lines, is a particularly useful technique for quantitative studies of the formation and growth of large grains.

EXPERIMENT

Polycrystalline iron foils were held at fixed temperatures between 200°C and 400°C as they were ion implanted. For each temperature, a foil was sequentially implanted with fixed doses of nitrogen ions (2.4×10^{17} ions/cm²) at each of three energies (80, 60, 40 keV). The foils were then examined by XRD and were examined in a scanning electron microscope (SEM) with subsequent elemental analysis by wavelength dispersive x-ray spectrometry (WDS) in the SEM.

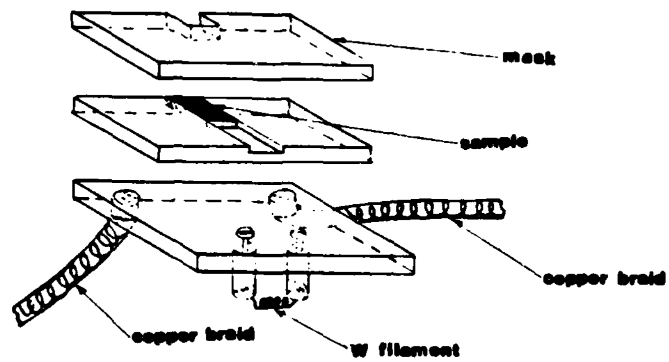

Figure 1
Schematic of stage for controlling target temperature during implantation. Stage is heated by 1-4 keV electrons from tungsten filament and cooled by copper braids.

The iron samples (99.999%, 25mm x 25mm x 2mm)[8] were mechanically polished to a 1 μm surface finish and chemically etched. The foils were clamped between thick copper plates (Figure 1). The entire assembly was heated by an electron gun and cooled by copper braids connected to a cold water bath. The front plate masked half the target area from the ion beam and provided an un-implanted control for each implantation. Target temperature was monitored by a chromel-alumel thermocouple mounted in a cylindrical cavity directly behind the target. An additional tantalum mask shielded all but the 25mm x 25mm target area from the ion beam to reduce beam heating and improve temperature stability.

The implantations were performed in a Zymet Z-100 ion implanter.[9] Magnetic mass selection is not available in this implanter so the targets were exposed to the raw output of the Freeman ion source. The ion source is known to produce roughly 70% N^+ and 30% N_2^+ ions although this was not monitored during these experiments. This implanter employs a bright pencil beam (≈ 4mm x 150mm) which is mechanically rastered across the target area. Typical peak current density during these experiments was 150 μA/cm² and typical average current density was 30 μA/cm². Typical base pressure in the vacuum chamber was 4×10^{-7} Torr and typical pressure during implantation was 1×10^{-5} Torr.

X-ray diffraction patterns were obtained on a Norelco diffractometer using intense Cu Kα radiation (λ = 1.5405 Å). Since this is well below the absorption edge in iron (1.7 Å), the diffracted radiation was filtered by a graphite crystal monochromator to suppress fluorescence from the iron. Patterns were obtained for the implanted and un-implanted sections of all samples between $12° \leq 2\theta \leq 72°$ and selected patterns were digitized for further analysis.

Microanalyses were performed using a JEOL JXA-840 scanning electron microscope equipped with two JEOL wavelength dispersive x-ray (WDS) spectrometers and a Tracor Northern 5500/5600 x-ray and image analyzer. Quantitative analysis for Fe, N, C, and O were performed by WDS and the data were corrected for atomic number, absorption and fluorescence using a Tracor Northern ZAF program.[10]

RESULTS AND DISCUSSION

Even at the lower temperatures, the XRD patterns show evidence of iron nitride phases (Figure 2). Peaks corresponding to γ'-Fe_4N appear in all patterns although these peaks are quite weak in the 200°C sample. This sample exhibits the (021) and (400) peaks of ζ-Fe_2N. In the 250° and 300° samples, we have tentatively attributed the 43.2° peak to the strongest reflection of the γ-austenite iron nitrogen solid solution even though Fe_3N should show a peak at roughly the same angle.[4] Over this temperature range, increasing temperature favors the formation of a nitride phase with stoichiometrically less nitrogen.

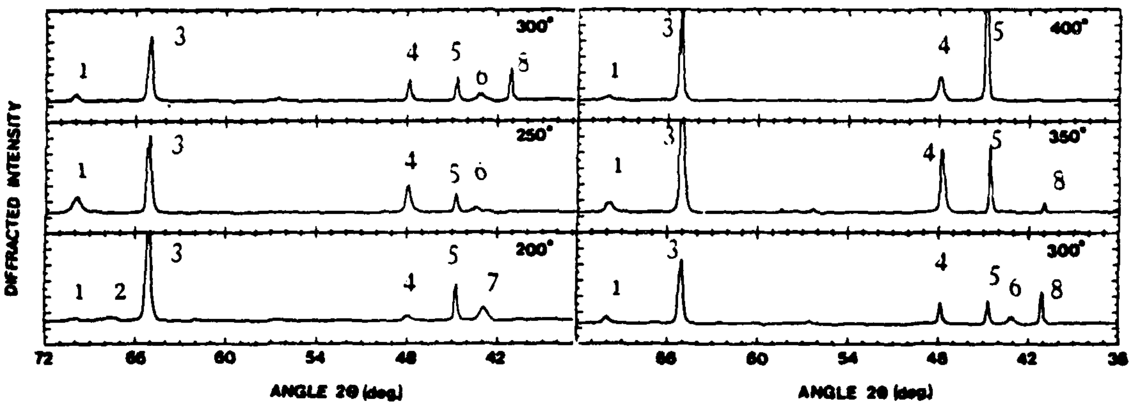

Figure 2
Phases with lower nitrogen content are favored at higher temperatures.
1) γ'-Fe_4N(220), 2) ζ-Fe_2N (400)
3) α-Fe (200), 4) γ'-Fe_4N(200)
5) α-Fe (110), 6) see text
7) ζ-Fe_2N (211), 8) γ'-Fe_4N(111)

Figure 3
Line intensities suggest an optimum temperature for growth
1) γ'-Fe_4N(200), 2) ζ-Fe_2N (400)
3) α-Fe (200), 4) γ'-Fe_4N(200)
5) α-Fe (110), 6) see text
8) γ'-Fe_4N(111)

At the higher temperatures, (Figure 3) the XRD patterns show predominately the γ'-Fe_4N phase which presents peaks with intensities comparable to the low angle α-Fe peak. The iron nitride lines tend to become stronger with increasing target temperature. However, in the 400°C sample, the nitride peaks appear somewhat weaker, perhaps indicating the existence of an optimum temperature for growth of Fe_4N grains during ion implantation. Another striking feature of these patterns is the change in relative intensity of the (110) and (200) α-Fe lines with increasing temperature. This might be interpreted as an annealing effect in our rolled polycrystalline samples. However, since the relative intensities of the iron line are also quite different between the implanted and unimplanted halves of each sample, we believe that the ion beam damage is itself inducing orientation changes in the near-surface iron.

Although it is difficult to image iron nitride grains directly in the SEM, distinct surface structures are visible (Figure 4). Furthermore, WDS performed in the SEM shows a dramatic decrease in nitrogen concentration with increasing temperature (Figure 5). This suggests that at higher target temperatures, nitrogen diffuses towards the surface and is lost either through sputtering or evaporation. Indeed, other work has shown that diffusion towards the surface strongly skews the final distribution of nitrogen implanted at high target temperatures.[11]

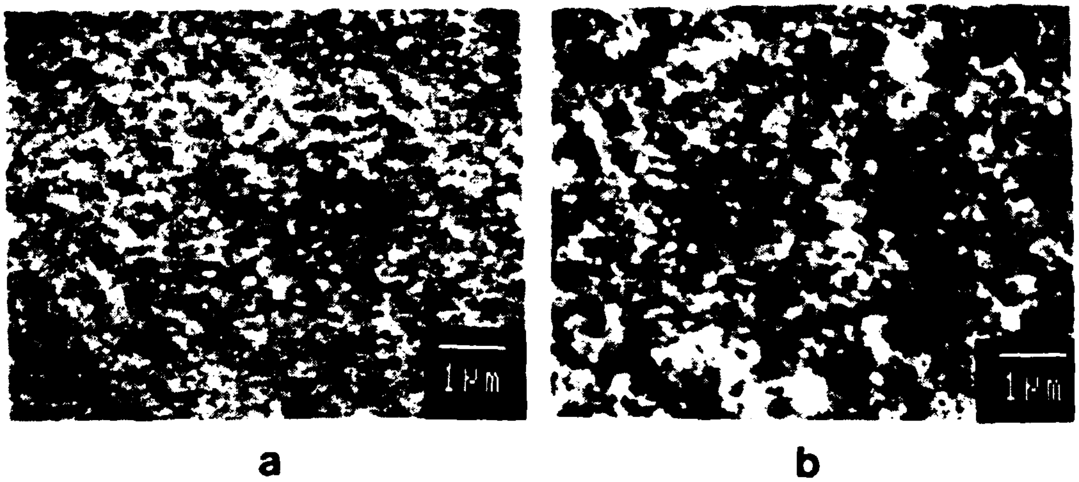

Figure 4.
SEM micrographs illustrate surface morphology of nitrogen implanted iron; 4a and 4b show surfaces implanted at 200°C and 350°C respectively.

Because of the increased defect density near the surface and instantaneous thermal spikes caused by the ion beam, dopant atoms should diffuse preferentially towards the surface. In addition, the target surface is severely eroded by sputtering under our experimental conditions. A calculated dopant profile,[12] using tabulated sputtering coefficients,[13] demonstrates the importance of this effect (Figure 6). In this calculation, dopant atoms are not allowed to diffuse but the target surface is eroded to a depth of more than 500 Å. Thus it is reasonable that a significant fraction of the dopant atoms are lost by sputtering after diffusion towards the surface and this mechanism retards the growth of crystalline grains at the higher target temperatures.

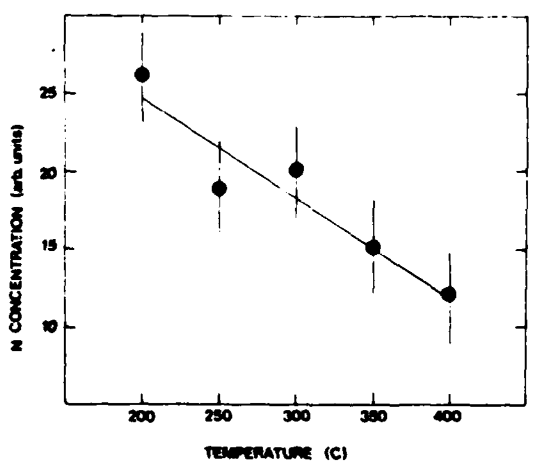

Figure 5.
WDS profiles indicate lower nitrogen concentration, due to out-diffusion, with increasing target temperature.

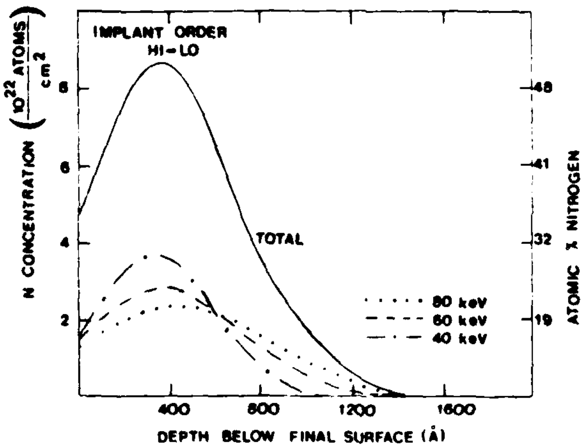

Figure 6.
Calculated dopant profiles, taking surface sputtering into account but ignoring dopant diffusion. Peak concentration is 50.3 atomic % at a depth of 372 Å. FWHM of the final distribution is 782 Å.

Increasing target temperature also increases the mobility of beam-induced target vacancies and other defects. There is evidence that crystalline grain growth during ion implantation is well correlated with the motion of vacancies introduced by the ion beam.[14,15] The competition between dopant diffusion to the surface and vacancy mobility should yield an optimum temperature for growth of iron nitride grains during ion implantation. Integral line breadths, measured from the (200) reflections of γ'-Fe_4N at various temperatures are consistent with this phenomenon (Figure 7). The narrowest lines, which are roughly as sharp as the underlying iron lines, indicate more perfect grains while broader lines show the effects of strain or small crystallite size.

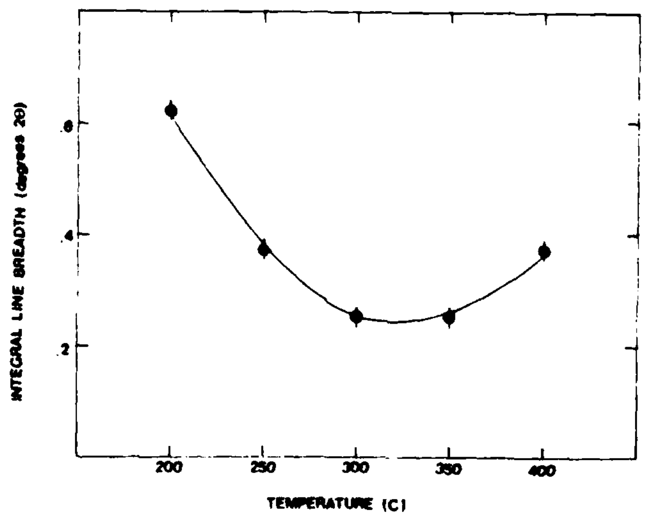

Figure 7.
X-ray line width data indicate the existence of an optimum temperature for iron nitride grain growth during ion implantation.

SUMMARY

At this preliminary stage, the well-resolved peaks in XRD patterns offer a quantitative picture of crystallite growth in thin ion implanted layers. The measured XRD patterns also contain information about orientation effects during implantation. Since XRD is not sensitive to small grains, complementary TEM information is required. The growth of grains large enough to produce sharp XRD lines is surprising but measured XRD patterns offer insight into the mechanisms involved and suggest an optimum target temperature for crystalline grain growth in nitrogen implanted iron.

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